A critical evaluation of thermal mass flow meters

S. A. Tison
National Institute of Standards and Technology, Gaithersburg, Maryland 20899

Many semiconductor processes require that stable and known flows of gas be delivered to the processing chamber. The thermal mass flow meter (TMFM) is used almost exclusively in the semiconductor industry for the admission of process gases. While TMFM's have been used in the semiconductor industry for over twenty years, much still remains to be understood about their behavior. The abundance of TMFM manufacturers that make instruments which are supposedly interchangeable complicates the use of TMFM's because the instruments generally have different designs and performance. While some attempt has been made via written standards to address the specifications of the instruments, these standards do not address all performance issues and cannot eliminate the systematic errors in the original manufacturers calibration of the TMFM's. Further, the TMFM's used to measure the process gases are generally calibrated with nitrogen and ''corrected'' for other gases, but the correction factors are not well understood and are of questionable reliability. It is also important to understand how the TMFM's perform under conditions that differ from the laboratory conditions where they were calibrated and the measurement errors that are introduced as a result of these different operating conditions. This article presents data on the performance of five low-flow TMFM's, from different manufacturers, with full scale ranges of 1.5×10^{-6}–3.7×10^{-6} mol/s (2–5 sccm). The manufacturers' calibration of the TMFM's with nitrogen as compared to the National Institute of Standards and Technology (NIST) measured values differed by up to 17%. Three of the five tested TMFM's were within the manufacturers' stated tolerance of $\pm 1\%$ of full scale. While some of the instruments' initial calibration was poor, all of the TMFM's were stable to within $\pm 1\%$ of full scale over the test interval of nine months. The gas correction factors for five gases (argon, helium, hydrogen, sulfur hexafluoride, and hexafluoroethane) were measured and compared to manufacturers' recommended values along with the temperature and flow dependence of the gas correction factors. Some of the gas correction factors agreed with the manufacturers' recommended values to within $\pm 1\%$ while others differed by as much as 13%.

I. INTRODUCTION

The measurement and control of gas flow are critical in many manufacturing processes. Semiconductor manufacturers, in particular, rely upon mass flow measurements for gas admission into processing tools or reaction vessels. The thermal mass flow meter (TMFM) is most prevalently used in the semiconductor industry. This meter senses the flow by measuring the thermal transfer between a heated tube wall and the gas stream. The TMFM's operate over a wide range of flow, 0.04–7.4×10^{-8} mol/s (5×10^4–0.1 sccm), and are suitable for use with most gases, including corrosives routinely used in the semiconductor industry. Flow measurement from 0.04–7.4×10^{-5} mol/s (5×10^4–100 sccm) has been routine for a number of years in the semiconductor industry, and the performance of thermal mass flow meters in this range has been investigated.[1] The use of TMFM's in the range of 7.4×10^6–7.4×10^{-8} mol/s (10–0.1 sccm) is becoming more prevalent, but their performance in this range is not well documented.

Due to the fact that TMFM's are often used with multiple gases or highly toxic gases, it is a common practice to calibrate the instrument with one gas, such as nitrogen, and employ ''generic'' correction factors to estimate the flow with other gases. Unfortunately, these correction factors are in-

strument specific and may vary by as much as 10% between instruments of different designs. Because the values of the correction factors may vary from 0.2 to 1.5, errors in the correction factors can add significant errors to measurements that rely upon them. Additionally, it has been suggested[2] that the correction factors may be a function of flow and not constant at all. Errors in the measured flow are also incurred when the temperature or pressure of the gas differs from their calibrated values. Manufacturers usually report an estimated uncertainty due to these effects, but the accuracy of these estimates is not known.

In this article we investigate the performance of TMFM's for a variety of gases and operating conditions that may be found in a manufacturing environment. The accuracy of the TMFM's manufacturers calibration with nitrogen and the range of variability between the recommended and actual correction factors for other gases are presented. The effects of operating conditions such as temperature, TMFM orientation, and pressure are presented. To accomplish this task, instruments from five manufacturers were chosen with full scale ranges between 1.5×10^{-6} and 3.7×10^{-6} mol/s (2 and 5 sccm). This flow range was selected because of its increasing importance to the semiconductor industry and the lack of

knowledge of the performance characteristics of TMFM's in this range.

II. TMFM's DESIGN AND OPERATING PRINCIPLES

The TMFM[2-4] senses flow by measuring the rate of heat transfer from a heated tube to the gas flowing inside the tube. The gas may be heated from ambient temperatures up to 100 °C inside the sensing tube of the TMFM. While designs between manufacturers vary, there are two measurement techniques that are commonly employed. The first, schematically shown in Fig. 1, is to provide a constant input power to a section of tubing and measure the temperature of the tube on both sides of the heated section. The flowing gas skews the temperature such that the downstream temperature is larger than the upstream value. This measured difference is linearly dependent upon mass flow to first order according to

$$\dot{m} = \frac{Q}{C_p(T)(T_{Gd} - T_{Gu})},\qquad(1)$$

where $C_p(T)$ is the temperature dependent molar heat capacity ($J\,mol^{-1}\,K^{-1}$), Q is the rate of heat transfer from the capillary wall to the gas ($J\,s^{-1}$), T_{Gd} is the gas temperature downstream of the heated capillary (K), T_{Gu} is the gas temperature upstream of the heated capillary, and $\dot{m}$ is the molar flow ($mol\,s^{-1}$). The second technique heats the tube by maintaining a constant temperature independent of flow. The amount of power required to maintain the constant tube temperature is then proportional to the mass flow in the tube.

Although the TMFM's output is normally linearly dependent on mass flow, nonlinearities may be introduced into the measurements in a number of ways. The gas temperature is normally measured by measuring the temperature of the capillary wall, which if different from the gas stream temperature will introduce errors. The rate of heat transferred from the capillary to the gas, Q, may be dependent upon the magnitude of the flow[2] and not only a function of the gas properties. The heat capacity of the gas may be temperature dependent, which may introduce nonlinearities into the flow measurement. Other heat loss mechanisms, such as radiative heat losses, may introduce additional nonlinearities.
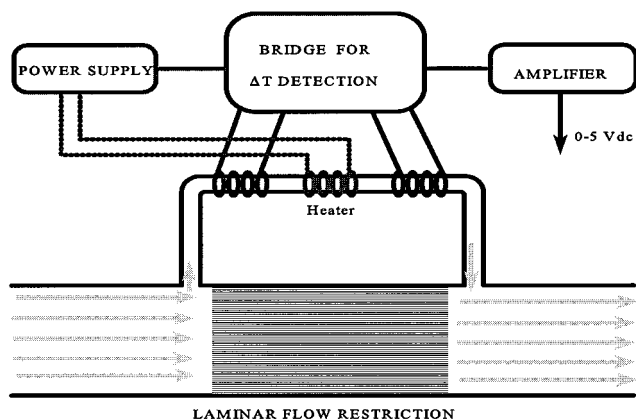
TMFM's are typically designed using very small stainless steel tubing with inside diameters varying from 0.25 to 1 mm and wall thickness minimized to lessen axial thermal losses in the tubing. The tube is wrapped with a number of heater windings which have a high resistance and a high temperature coefficient of resistance. This allows the heater to become a temperature sensor as well as a heat source. TMFM's are designed so that the flow is laminar with maximum flows through the tube less than 7.4×10^{-6} mol/s (10 sccm). Larger flow TMFM's are constructed by splitting the flow with a channel which bypasses the sensor. The particular attributes of the TMFM's used in this study, including manufacturers' specifications, are given in the Appendix. It is National Institute of Standards and Technology (NIST) policy to identify instruments only by their generic specifications.

III. APPARATUS AND MEASUREMENT TECHNIQUE

The TMFM's were calibrated by direct comparison with a NIST constant-pressure primary standard flowmeter. The flowmeter generates and measures flow by advancing a piston of known volume into a vessel at a rate such that the pressure in the vessel remains constant while the gas escapes through an attached leak valve into a vacuum system. The flowmeter generates and measures flow over a range of $1 \times 10^{11} - 5 \times 10^{-6}$ mol/s ($1 \times 10^{-5} - 7$ sccm) and is described in detail elsewhere.[5] The uncertainty of the flowmeter in the range of testing, $5 \times 10^{-8} - 5 \times 10^{-6}$ mol/s, is $\pm0.1\%$, representing two standard deviations. The comparison of the flows between the TMFM's and the NIST piston flowmeter was effected in the following manner using spinning rotor gauges (SRG's). The measurement apparatus is shown schematically in Fig. 2. The pressure readings of two SRG's in the vacuum chamber downstream of the piston flowmeter are recorded with no flow. The flow from the piston flowmeter is directed into the vacuum chamber and evacuated through a 1 cm orifice, which has a stable conductance or "throughput." The equilibrium pressure above the orifice is measured and recorded by two SRG's. The flow is changed and this process is repeated over the flow range of interest of $7.4 \times 10^{-8} - 4 \times 10^{-6}$ mol/s (0.1–5 sccm) for nitrogen. A correlation between the known flow from the piston flowmeter and the pressure measured by the SRG's is then determined from this data. For nitrogen the measured pressures ranged from 0.02 to 1 Pa. To first order the relationship between

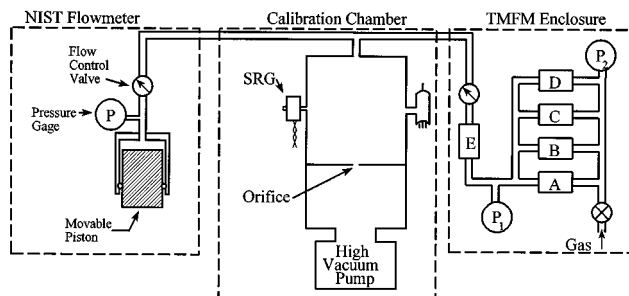FIG. 1. Schematic of a typical thermal mass flow meter.

FIG. 2. Schematic of the NIST system for calibrating TMFM's.

flow and the observed pressure is linear, but due to small deviations from molecular flow through the orifice and possible SRG nonlinearities, a second order polynomial was used. This process was repeated for argon and sulfur hexafluoride. The calculated total flow uncertainty using this technique is ±0.32%, representing two standard deviations. This uncertainty is dominated by instabilities of the SRG's which are estimated to be 0.3% during the testing period.

The tested TMFM's, which are labeled $A-E$ to preserve the manufacturers' anonymity, are described by their generic specifications and operating principles in the appendix. TMFM's $A-D$ were capable of measuring and controlling flows, while TMFM E had no flow controlling capability. The flow control point of TMFM's $A-D$ was set via an analog voltage, 5 V dc maximum, which is proportional to the generated flow. All of the TMFM's provided an analog voltage output, 5 V dc maximum corresponding to the full scale output, which is proportional to the measured flow. This voltage was measured with a high accuracy digital voltmeter and converted to the appropriate flow.

For testing, the TMFM's were mounted in a temperature controlled enclosure that was normally operated at 25 °C (±0.5 °C). TMFM's $A-D$ were mounted parallel to one another, and TMFM E was mounted in series with TMFM's $A-D$ (see Fig. 2) because it lacked flow controlling capability. The volume between TMFM E and the others was minimized by using 0.4 cm inside diameter tubing with small lengths. Downstream of TMFM A was a 0.1 MPa full scale pressure gauge (P_1), TMFM E, and a variable conductance valve that was adjusted to achieve the desired pressure downstream of TMFM E (50–75 kPa). Upstream of the TMFM's was a gas handling system, a 1 μm filter, and a 0.15 or 1 MPa full scale pressure gauge (P_2).

To calibrate the TMFM's, they were first "zeroed" in the following manner. The upstream pressure of TMFM's $(A-E)$ was set to 0.1 MPa with the regulating valves fully open. A valve upstream of TMFM's $A-D$ was closed and the variable conductance valve downstream of TMFM E was closed so that there was no gas flow. The readings of the TMFM's were monitored until the signals equilibrated and the "zero" readings were recorded. After "zeroing," the flow control valves for TMFM's $A-D$ were closed and the valves upstream and downstream of the TMFM's opened to allow gas to flow. The flow control point for TMFM A was then changed via a remote analog set point with a programmable power supply. The flow was directed into the vacuum chamber and evacuated through the 1 cm orifice. Upon equilibrium, the outputs of TMFM A and E were recorded, along with the observed pressure above the 1 cm orifice (measured with two SRG's). This process was repeated at increments of 10% of the instruments' full scale to 100% full scale and at 10% decrements down to 0. The actual flow was computed using the observed pressure readings of the SRG's and the previously described relationship between these values and the flow. This process was repeated with TMFM's B, C, and D.

IV. RESULTS

A. Nitrogen calibration

The TMFM's were first calibrated with nitrogen at NIST within 3 months of the manufacturers' calibration. The results of this calibration are shown in Fig. 3 for TMFM's $A-E$. Each data point in Fig. 3, as well as in subsequent figures and tables, represents an average flow reading taken over a 5 min interval. TMFM's A, C, and E were within their manufacturers stated uncertainties of ±1% full scale. The results for TMFM A indicate that the manufacturer's calibration at the full scale value was in very good agreement with the NIST value, but the deviations increased for lower flow rates to a maximum of 0.6% of full scale (1.2% of reading at 50% of full scale). TMFM C gives results that are systematically low by 0.4% from the NIST measured values. TMFM E has no significant systematic trends within the uncertainties of these measurements. TMFM B exhibited systematic differences ranging from −10% at 70% of full scale to −17% at 10% of full scale. TMFM D exhibited a systematic offset that varied from 8.5% to 6.8% higher than the measured NIST values. The differences between the TMFM's indicated flow and the NIST measurements are most likely to be due to systematic differences between the manufacturers' standards and NIST standards used for their calibration and not due to instabilities in the TMFM's themselves. This assertion is substantiated by the long-term stability of the TMFM's which is detailed in Sec. IV F.

B. Correction factors

The gas correction factor is defined as the indicated flow of the test gas (sccm) divided by the equivalent nitrogen flow (sccm). The NIST measured nitrogen flow was used in place of the manufacturers' predicted values to minimize the effects of systematic errors introduced by the manufacturers'
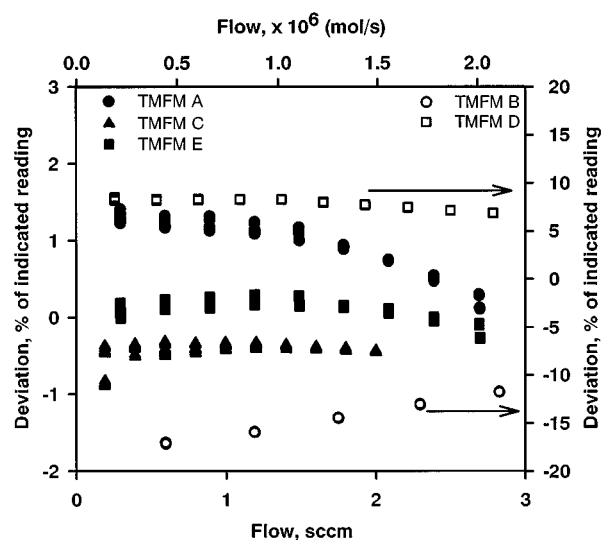

FIG. 3. Deviations of the manufacturers' nitrogen calibrations of TMFM's from the NIST measured values. TMFM's A, C, and E data correspond to the left axis and TMFM's B and D data correspond to the right axis.

TABLE I. Correction factors (CFs) for TMFM's using argon gas relative to a nitrogen calibration. (FS is an abbreviation for full scale; Man. is an abbreviation for manufacturer.)

TMFM	CF (25% FS)	CF (50% FS)	CF (75% FS)	CF (FS)	Average	Man. value	Difference (avg.-Man. %)
A	1.423	1.423	1.426	1.432	1.426	1.443	−1.19
B	1.413	1.423	1.430	1.437	1.426	1.400	1.82
C	1.427	1.422	1.425	1.430	1.426	1.396	2.15
D	1.417	1.417	1.423	1.425	1.421	1.398	1.62
E	1.415	1.417	1.418	1.420	1.418	1.370	3.39
Average	1.419	1.420	1.424	1.429	1.423	1.401	1.55

calibration upon the measured correction factors. The measured correction factors between nitrogen and the test gases are given in Tables I–V at 25%, 50%, 75%, and 100% of full scale (FS) for the test gases.

1. Argon

The results of the calibration with argon are given in Table I. In general, the correction factor is found to be a function of flow and to first order to generally increase linearly with increasing flow. The value of the change varies from 0.2% for TMFM E to 1.7% for TMFM B, with the other TMFM's having intermediate values. The last row of Table I contains the computed correction factors of the TMFM's averaged over all meters. From these data it can be surmised that the correction factor increases with increasing flow with an average change of 0.7% over the given range. The manufacturers' (Man.) reported values, column 7, differed considerably from the measured values. The maximum deviations between the manufacturers' recommended values and the observed values ranged from 1.6% to 3.6%. These deviations are not surprising, as most manufacturers stipulate that the uncertainties of the correction factors are on the order of a few percent. What is surprising is the agreement of the observed correction factors between different manufacturers. The average correction factor for the TMFM's (averaged over all flows), given in column 6, ranged from 1.426 to 1.418 with an average of 1.423, which represents less than 0.6% maximum variation. Use of the grand average value (average of all TMFM's) of 1.423 results in deviations from the observed values of less than 1%.

2. Helium

The results of the calibration with helium are given in Table II. In general, the correction factor is found to be a function of flow and to first order to decrease linearly with increasing flow. The value of the change varies from no change for TMFM B to −1.2% for TMFM D, with the other TMFM's having intermediate values. The last row of Table II contains the computed correction factors of the TMFM's averaged over all meters. From these data it can be surmised that the correction factor decreases with increasing flow with an average change of −0.6% over the given range. The manufacturers' (Man.) reported values, column 7, differed considerably from the measured values. The maximum deviations between the manufacturers' recommended values and the observed values ranged from 0.3% to 3.1%. The average correction factors for the TMFM's A, B, C, and E (averaged over all flows), given in column 6, ranged from 1.432 to 1.446 with an average of 1.438, which represents less than 0.6% maximum variation. TMFM D exhibited a significantly larger correction factor with an average value of 1.475, 2.5% higher than the average value for the other TMFM's.

3. Sulfur hexafluoride

The results of the calibrations with sulfur hexafluoride are given in Table III. The average correction factor as given in the last row varies little with flow. Individually, the TMFM's show variations with flow from no change to a maximum change of −1.8%. The average correction factor for individual TMFM's, given in column 6, ranged from 0.268 to 0.281, which represents a maximum variation of 9%. While

TABLE II. Correction factors (CFs) for TMFM's using helium gas relative to a nitrogen calibration.

TMFM	CF (25% FS)	CF (50% FS)	CF (75% FS)	CF (FS)	Average	Man. value	Difference (avg.-Man. %)
A	1.440	1.435	1.435	1.430	1.435	1.390	3.13
B	1.440	1.440	1.440	1.440	1.440	1.444	−0.28
C	1.452	1.450	1.442	1.442	1.446	1.434	0.83
D	1.483	1.480	1.470	1.465	1.474	1.445	1.97
E	1.434	1.432	1.431	1.430	1.432	[a]	
Average	1.450	1.447	1.444	1.441	1.445	1.428	1.17

[a]Manufacturer did not give a value in the manual.

TABLE III. Correction factors (CFs) for TMFM's using sulfur hexafluoride gas relative to a nitrogen calibration.

TMFM	CF (25% FS)	CF (50% FS)	CF (75% FS)	CF (FS)	Average	Man. value	Difference (avg.-Man. %)
A	0.277	0.276	0.275	0.274	0.275	0.275	0.00
B	0.267	0.267	0.268	0.270	0.268	0.270	−0.75
C	0.273	0.273	0.272	0.272	0.272	0.275	−1.10
D	0.261	0.258	0.257	0.256	0.258	0.260	−0.78
E	0.284	0.282	0.280	0.279	0.281	0.284	−1.07
Average	0.272	0.271	0.270	0.270	0.271	0.273	−0.74

correction factors of TMFM's varied widely, the manufacturers' recommended values are in good agreement with the measured average correction factors listed in column 6. The maximum deviations between the manufacturers' recommended values and the observed values ranged from 1.8% to −0.7%. Use of a grand average correction factor (an average of all TMFM correction factors) in place of the measured value for the instrument, while not introducing considerable error for the case of argon, would lead to significant errors for sulfur hexafluoride.

4. Hexafluoroethane

The results of the calibrations with hexafluoroethane are given in Table IV. The average correction factor as given in the last row varies little with flow. Individually, the TMFM's show variations with flow from +0.4% to −0.3%. The average correction factor for individual TMFM's, given in column 6, ranged from 0.2506 to 0.2713, which represents a maximum variation of 8%. The correction factors of TMFM's vary widely and the manufacturers' recommended values are in poor agreement with the measured values of the average correction factors listed in column 6. The maximum deviations between the manufacturers' recommended values and the observed values ranged from −13.8% to 1.7%. The average disagreement between the manufacturers' recommended values and the average measured values was 7.7%.

5. Hydrogen

The results of the calibration with hydrogen are given in Table V. In general, the correction factor is found to be a function of flow and to first order to decrease linearly with increasing flow. The value of the change varies from no change for TMFM C to 5.3% for TMFM B, with the other TMFM's having intermediate values. The last row of Table V contains the computed correction factors of the TMFM's av-

eraged over all meters. From these data it can be surmised that the correction factor decreases with increasing flow with an average change of −2.2% over the given range. The manufacturers' (Man.) reported values, column 7, differed considerably from the measured values. The maximum deviations between the manufacturers' recommended values and the observed values ranged from −12.9% to −7.8%. The average correction factors for the TMFM's A, B, C, D, and E (averaged over all flows), given in column 6, ranged from 1.108 to 1.160 with an average of 1.1316, which represents a 2.5% maximum variation.

6. Discussion of gas correction factors

It is interesting to note that the correction factors for gases such as argon and helium exhibit great similarity between TMFM's from different manufacturers while for sulfur hexafluoride and hexafluoroethane there are large measured differences. It is not coincidental that the agreement for gases such as argon and helium is good. The heat capacity for these gases has small temperature dependencies,[6,7] less than 0.01% per °C, while the heat capacity for hexafluoroethane[7] changes by 0.18% per °C. To first order the correction factors are equal to the ratio of the molar heat capacities of the gases to the molar heat capacity of nitrogen. Differences in the operating temperatures of the sensing tubes for different TMFM's will cause differences in the correction factors for gases with large heat capacity temperature coefficients while causing relatively small changes for gases that have small heat capacity temperature coefficients. Most manufacturers list the heat capacity of gases next to the gas correction factors in the manuals. Unfortunately, these values are listed at reference temperatures of 0 or 25 °C while the TMFM's sensing tubes operate at 50–100 °C. The correction factors predicted from the ratio of the heat capacities[6] of the gases are compared to the average measured values for TMFM

TABLE IV. Correction factors (CFs) for TMFM's using hexafluoroethane gas relative to a nitrogen calibration.

TMFM	CF (25% FS)	CF (50% FS)	CF (75% FS)	CF (FS)	Average	Man. value	Difference (avg.-Man. %)
A	0.2665	0.2665	0.2667	0.2670	0.2667	0.230	13.76
B	0.2590	0.2595	0.2600	0.2600	0.2596	0.240	7.55
C	0.2625	0.2627	0.2630	0.2635	0.2629	0.247	2.46
D	0.2510	0.2503	0.2507	0.2502	0.2506	0.255	−1.76
E	0.2718	0.2711	0.2711	0.2712	0.2713	0.240	11.54
Average	0.2622	0.2620	0.2623	0.2624	0.2622	0.242	7.70

TABLE V. Correction factors (CFs) for TMFM's using hydrogen gas relative to a nitrogen calibration.

TMFM	CF (25% FS)	CF (50% FS)	CF (75% FS)	CF (FS)	Average	Man. value	Difference (avg.-Man. %)
A	1.1400	1.1350	1.1250	1.1220	1.1305	1.01	10.66
B	1.1400	1.1100	1.1000	1.0800	1.1075	1.021	7.81
C	1.1400	1.1400	1.1400	1.1400	1.1400	1.015	10.96
D	1.1900	1.1600	1.1500	1.1400	1.1600	1.024	11.72
E	1.1190	1.1200	1.1200	1.1200	1.1198	1.010	9.80
Average	1.1458	1.1330	1.1270	1.1204	1.1316	1.016	10.22

$A - E$ in Table VI. The predicted values for argon and helium are slightly less than the average measured values while for SF_6 and C_2F_6 all the observed values fall within the predicted values for the average gas temperatures between 50 and 100 °C. From these data, it is hypothesized that the differences in correction factors between TMFM's from different manufacturers are mainly due to differences in the average temperature of the gas in the sensing tube.

The temperature dependence of the heat capacity can also cause gas correction factors to be flow dependent due to the change in the average gas temperature for different flows. The flow dependence of the gas correction factor can also be caused by changes in the heat transfer process which is gas species dependent. This problem is more fully discussed by Hinkle and Mariano.[2] It should be noted that, for commercially available TMFM's, which typically have a maximum gas flow through the sensing tube of 10 sccm, this latter effect is typically limited to a few percent for most gases.

C. Orientation effects

Because TMFM's sense mass flow by measuring heat transfer to the gas, anything that influences the heat transfer process will influence the measurement of the mass flow. Mounting of the TMFM's affects the heat transfer by changing the amount of natural convection that takes place. This will most noticeable result in zero changes, particularly when the sensing tube is rotated from perpendicular to parallel to gravity. Changing the orientation of the tested TMFM's from the normal position, perpendicular to gravity (orientation 1), to an orientation where the sensing tube is parallel to gravity and the direction of flow is coincident with gravity (orientation 2) resulted in zero changes as large as 0.4% of full scale. The effect of changing the TMFM's orientation from orientation 1 to 2 upon the zero and the span of the TMFM's was determined with nitrogen and hexafluoroethane.

1. Zero

The measured zero changes for nitrogen varied from 0.02% of full scale for TMFM A to 0.15% of full scale for TMFM C. The measured zero changes for hexafluoroethane varied from no change for TMFM's C and E to 0.44% of full scale for TMFM D. None of the manufacturers gave specifications for the change in zero due to changes in the TMFM orientation. However, the manufacturers do recommend that the TMFM be re-zeroed after an orientation change.

2. Span

The change in the nitrogen orientation correction factor, the ratio of the flow for orientation 2 to that for orientation 1, is shown in Fig. 4. The data are compensated for zero changes caused by the change in orientation. As can be seen in Fig. 4, no significant change in the measured flow was observed. It would be anticipated that a change in orientation from 1 to 2 would significantly affect the zero of the TMFM because of the induced free convection in the sensing tube as was observed in the previous section. Changes in the span of the instruments would be caused by second order effects which could be the result of induced turbulence in the sensing tube due to the free convection. The data show these effects, if present, to have a negligible effect on the performance of the TMFM's. The hexafluoroethane orientation data show similar results. The relative unimportance of the TMFM orientation on the span could be predicted by examining the magnitude of the ratio between the buoyant forces, induced by changing from orientation 1 to 2, and those due to the pressure gradient. This ratio, commonly referred to as the Richardson number[7] (Ri), is typically much less than 0.1 for normal operation of the TMFM's which reflects the relatively small importance of buoyant forces. The small importance of the orientation effect is also recognized by the ma-

TABLE VI. Comparison of the measured gas correction factors (CF) for TMFM's with those predicted from the ratio of the gas specific heats at the given reference temperature. [Calculated (Cal.).]

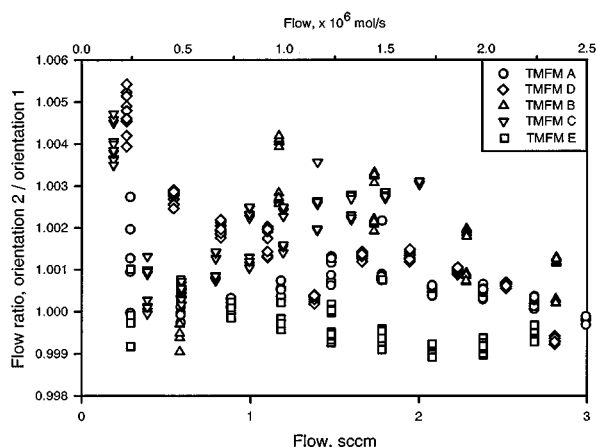| Gas	TMFM A (CF)	TMFM B (CF)	TMFM C (CF)	TMFM D (CF)	TMFM E (CF)	Cal. CF (50 °C)	Cal. CF (100 °C)
Helium	1.4350	1.4400	1.4460	1.4740	1.4320	1.413	1.417
Argon	1.4260	1.4260	1.4260	1.4210	1.4180	1.395	1.397
SF_6	0.2750	0.2680	0.2720	0.2580	0.2810	0.285	0.257
C_2F_6	0.2667	0.2596	0.2629	0.2506	0.2513	0.262	0.238

FIG. 4. Flow ratio of the TMFM's when calibrated in orientation 2, sensing tube perpendicular to gravity, from their calibration in orientation 1, sensing tube parallel to gravity with the flow coincident with gravity.

jority of TMFM manufacturers who typically specify that the maximum change in sensitivity after appropriate re-zeroing is less than ±0.5%.

D. Pressure effects

The sensitivity of TMFM's depend slightly on the upstream pressure. Manufacturers typically give uncertainties due to changes in pressure of approximately 0.75% per MPa. Increasing the upstream pressure (for a given temperature and mass flow) will cause the average velocity in the sensing tube to decrease. This velocity change may cause small changes in the sensitivity of the TMFM due to changes in the amount of heat transfer between the heated capillary and the gas.[7] The pressure effect was measured by determining the sensitivity of the TMFM's with nitrogen with upstream pressures of 100 and 250 kPa. All five TMFM's showed no significant change in sensitivity; sensitivity changes were less than 0.1%. This may not hold true for other gases, especially gases that are near their critical point and for which changes in the specific heat due to small pressure changes may be significant.

TABLE VII. TMFM's zero change due to an operating temperature change from 25 to 35 °C.

| | TMFA's | | | | |
	A	B	C	D	E
Measured change (% FS)	−0.1	−0.15	−0.15	0.10	0.10
Manufacturer specification (% FS)	±0.75	a	a	a	±0.40

[a]Values are not given by the manufacturer.

E. Temperature effects

Manufacturers typically specify the effect of temperature upon the TMFM zero and span. Although the manufacturers may recognize that these effects are gas specie dependent, usually only one generic specification is given. This generic specification may overestimate or underestimate these effects for a particular test gas. The temperature effects were measured by calibrating the TMFM's at 25 °C and then subsequently calibrating them at 35 °C and then again at 25 °C for the test gas of interest.

1. Zero

The change in the zero readings for the TMFM's when operated at 35 °C from the value when operated at 25 °C is shown in Table VII as a percent change of full scale. The measured changes are much lower than the values specified by the manufacturers of TMFM's *A* and *E*. TMFM *C* has an autozeroing feature that is designed to automatically compensate for this change. Manufacturers of TMFM's *B* and *D* did not report the zero temperature coefficient of their instrument. It should be emphasized that the zero temperature coefficients were measured with the TMFM's in orientation 1 and will differ in other orientations.

2. Span

The major contributors to the span temperature dependence of TMFM's are electronic changes and changes in the heat capacity of the gas. To separate these two effects, the temperature dependence was first measured with both helium and argon gases. The heat capacities of these gases change

TABLE VIII. Measured span temperature coefficient (temp. coeff.) for TMFM's given as a percent of reading by changing the inlet gas temperature from 25 to 35 °C.

TMFM	Electronic temp. coeff. (% °C^{-1}×10)	Nitrogen temp. coeff. (% °C^{-1}×10)	SF$_6$ temp. coeff. (% °C^{-1}×10)	C$_2$F$_6$ temp. coeff. (% °C^{-1}×10)	Man. temp. coeff. (% °C^{-1}×10)
A	−0.30	−0.30	−1.50	−1.80	0.75
B	+1.80	0.00	−1.40	−1.80	1.00
C	+2.00	+0.20	−0.90	−1.20	a
D	+0.60	+0.20	−1.40	−1.80	a
E	−0.10	+0.30	−0.90	−1.30	0.80
Average	+0.80	0.08	−1.22	−1.58	···

[a]Manufacturers state that the TMFM's are temperature compensated.

less than 0.04% over the tested temperature interval, so that changes in the TMFM's span or sensitivity are due solely to electronic changes. The temperature dependence of nitrogen, sulfur hexafluoride, and hexafluoroethane were subsequently measured. The temperature dependence measurement results are given in Table VIII. The electronic temperature dependence has been subtracted from the measured temperature dependence of the TMFM's to observe the temperature coefficient which is due only to changes in the gas properties. The measured electronic temperature coefficient values are lower than the manufacturers' values for TMFM's *A* and *E* for whom specifications were given; see column 6 Table VIII. TMFM *B* changed by −1.8% of full scale which is almost double the manufacturers specified tolerance of 1% of full scale. The manufacturers of TMFM's *C* and *D* claim that their instruments are temperature compensated and do not list a temperature coefficient. This compensation was not apparent as the instruments exhibited temperature coefficients similar to those not making this claim. The heat capacity of nitrogen changes by 0.06% over the tested temperature range. The average measured change, shown in the last row of Table VIII, is 0.08% which compares favorable due to the change in heat capacity. In practicality this is too small of a change to be significant with TMFM's which claim a repeatability of ±0.2%. The change in heat capacity of sulfur hexafluoride of 1.7% compares favorably to the measured TMFM temperature coefficients. The agreement is best for TMFM's *A*, *B*, and *D* which operate with a constant input power. For these instruments a change in the input gas temperature should correspondingly change the average gas temperature in the sensing capillary. TMFM's *C* and *E* use a constant temperature sensing technique. For these instruments a change in the input gas temperature will result in a smaller change in the average gas temperature than for the other instruments. The amount of this difference will be dependent upon the inlet gas temperature and the temperature at which the capillary is maintained. The change of 1.87% in heat capacity of hexafluoroethane over the tested temperature range compares favorably to the measured TMFM temperature coefficients. Again, the agreement is best for TMFM's *A*, *B*, and *D* which operate with a constant input power. TMFM's *C* and *E* have smaller temperature coefficients which is due to their different sensing technique as explained previously for the sulfur hexafluoride case.

F. Stability

In addition to quantifying the uncertainties in the TMFM's, it is desirable to know the stability of the instruments over time. All of the TMFM's were calibrated repeatedly over a nine month period with nitrogen to determine their stability. Between tests the power was maintained to the TMFM's and they remained in the same physical location without exposure to corrosive gases. The TMFM's were periodically cycled from 25 to 35 °C and used with all of the calibration gases discussed in this article. The stability of the TMFM's are measured in changes of their span, or sensitivity, and zero.

1. Span stability

The results of the nitrogen calibrations expressed as a deviation in the TMFM's span from the first calibration at their full scale value are shown in Fig. 5. One test sequence with TMFM *D* gave results that deviated by up to 2% from the initial calibration. These data were deemed to be anomalous and not included in the summarized results because the instrument subsequently returned to its original calibration and never exhibited this behavior in any later tests. From Fig. 5 it can be seen that all of the TMFM's remained within ±1% of full scale from their first calibration. In fact, all of the TMFM's nitrogen calibrations remained within ±1% of reading over a range from 10% to 100% of full scale over the nine month testing period. TMFM *A*, although it had excellent measurement stability, had difficulty controlling flows below 25% of its full scale after two months of operation. The results in Fig. 5, when combined with the results shown in Fig. 3, indicate that TMFM's *A*, *C*, and *E* remained within their manufacturers' uncertainties of ±1% full scale during the testing period, whereas TMFM's *B* and *D* remained outside their prescribed manufacturers' uncertainties. Repeated determinations of the gas correction factors for helium, argon, hydrogen, sulfur hexafluoride, and hexafluoroethane showed no significant changes, less than 0.2%, in the correction factors with time for all of the tested TMFM's.

2. Zero stability

While the sensitivity for the TMFM's showed very good repeatability with time, the stability of the zero reading exhibited significant changes for some of the TMFM's. TMFM's *B* and *E* were the most stable, maintaining their zero to within ±0.04% of full scale over the nine month testing period for the standard testing conditions. TMFM *A* showed a maximum deviation of ±0.08% of full scale while TMFM's *C* and *D* exhibited changes as large as ±0.4% of
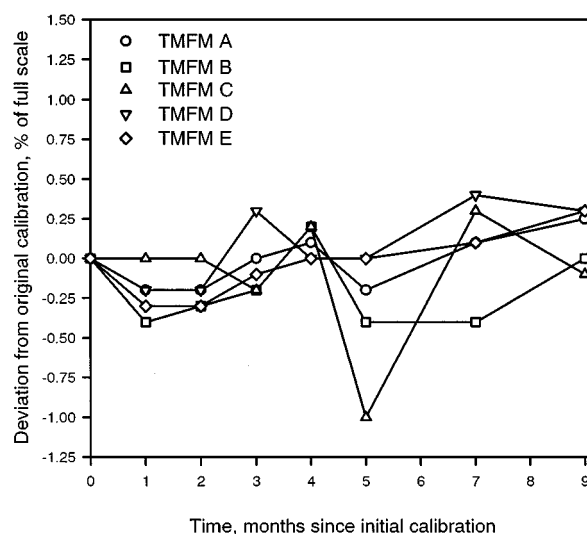
FIG. 5. Stability of the TMFM's over a nine month period relative to their first NIST nitrogen calibration.

full scale during the testing period. This zero stability reflects operation of the TMFM's with nitrogen in orientation 1 with the temperature maintained at 25±0.5 °C.

G. Low flow performance

The TMFM manufacturers specify that the instruments measure and control flow down to 2% of their full scale value. All of the TMFM's meet this specification when new, but TMFM A would no longer control flows below 25% of its full scale after two months of operation. In general, the TMFM's were able to generate flows stable to within ±0.2% of reading over this range when averaged over a five second interval. Additionally, long-term stability of the calibration (nine month period) was within ±1% of reading for this range when the TMFM's were appropriately zeroed.

V. SUMMARY

Three out of the five TMFM's performance were in reasonable agreement with the manufacturers specifications of ±1% of full scale when operated with nitrogen with the exception of the manufacturers' calibration of TMFM's B and D which were significantly outside of their prescribed uncertainty. The TMFM's calibrations were stable over a nine month period to within ±1% of reading over the range of 2%–100% of their full scale when appropriately zeroed. The gas correction factors for argon were almost identical (1.423) for all of the TMFM's tested and the temperature dependence of the gas correction factor was negligible. The gas correction factor for helium differed from the manufacturers' reported values by up to 3%, but was nearly identical for four of the five tested TMFM's. The temperature dependence of the gas correction factor for helium was negligible. The gas correction factor for sulfur hexafluoride differed from the manufacturers' values by up to 1% with a maximum difference between the measured correction factors of 9%. The average temperature dependence of the correction factor for sulfur hexafluoride was measured to be -0.12% °C^{-1} which is in reasonable agreement with the expected change due to the change in the heat capacity of the gas. The gas correction factor for hexafluoroethane differed from the manufacturers' values by up to 11% with a maximum difference between the measured correction factors of 8%. The average TMFM's temperature dependence of the correction factor for hexafluoroethane was measured to be -0.16% °C^{-1} which is in reasonable agreement with the expected change due to the change in the heat capacity of the gas. The gas correction factor for hydrogen differed from the manufacturers' values by up to 13% with a maximum difference between the measured correction factors of 5%.

Some of the gas correction factors of the TMFM's showed flow dependences which were largest for helium and hydrogen. Orientation changes were found to change the TMFM's zero indication by less than 0.44% of full scale and the effect on the TMFM's span was negligible. Changes in the upstream pressure were found to have a negligible effect for nitrogen, but may be significant for gases with low vapor pressures.

Most commercial TMFM's in use are of a higher flow range than those in this study and are not fully shunted. In these flowmeters, a large percentage of the flow is directed through a laminar bypass and not through the sensing tube. Although many of the effects in this study will be applicable to these types of flowmeters, there is probably not a direct correspondence. The addition of the bypass section will tend to complicate the relationship between the changes in gas properties and the resulting changes in the measured flow. This will occur primarily due to the temperature dependence of the gas viscosity. The temperature dependence of the gas viscosity will change the correction factors for some gases from those measured with TMFM's that have no bypass section. Additionally, the temperature dependence of the viscosity may introduce a larger flow dependence in the gas correction factors.

VI. CONCLUSIONS

Five TMFM's with full scale ranges of 1.5×10^{-6}–3.7×10^{-6} mol/s (2–5 sccm) were investigated to determine their uncertainties with nitrogen gas, their correction factors with other gases, and the effect of variable operating conditions upon their performance. It was found that three of the five TMFM's were within the manufacturers' stated uncertainty of ±1% of full scale. Two TMFM's were well beyond their stated uncertainty, one by as much as 17%. The measured gas correction factors for the test gases of argon, helium, hydrogen, sulfur hexafluoride, and hexafluoroethane deviated from the manufacturers' recommended values by as much as 15%. The measured temperature coefficients for the TMFM's were gas species dependent, although predictable to some level. Orientation and upstream pressure changes had little effect on the calibration of the TMFM's if the instruments were appropriately zeroed. All of the tested TMFM's were found to be repeatable to within ±1.0% of reading over a nine month period.

APPENDIX

TMFM's A–D are capable of measurement and control whereas TMFM E is a meter only. The manufacturers' description and specifications for the TMFM's are given in Table AI. Column 5 of Table AI refers to the geometry of the sensing tube. Those TMFM's that are indicated as having a bypass have a sensing capillary that bypasses a totally shunted (blocked) main flow section. Those TMFM's that are indicated as not having a bypass flow the gas through a straight section of the sensing capillary.

TABLE AI. Characteristics of the five tested TMFM's. The description and specifications are those given by the manufacturers in the instruments manual (FS is an abbreviation for full scale; Rdg is an abbreviation for reading).

TMFM	Full scale (sccm)	Control valve type	Sensing technique	Bypass section	Repeatability specification	Zero temp. sensitivity (per °C)	Span temp. sensitivity (per °C)
A	3	Solenoid	Constant power	Yes	0.25% of FS	0.075% of FS	0.05% of FS
B	5	Solenoid	Constant power	Yes	0.2% of FS	Not reported	0.1% of FS
C	2	Piezoelectric	Constant temp.	No	0.2% of FS	Not reported	Not reported
D	3	Solenoid	Constant power	Yes	0.2% of FS	Not reported	Not reported
E	2	None	Unknown	No	0.2% of FS	0.04% of FS	0.08% of Rdg

[1] J. Riddle and J. Hardy, SEMATECH Report No. 9402373A-XFR (1994).
[2] L. D. Hinkle and C. F. Mariano, J. Vac. Sci. Technol. A **9**, 2043 (1991).
[3] K. Komiya, F. Higuchi, and K. Ohtani, Rev. Sci. Instrum. **59**, 477 (1988).
[4] C. W. Hawk and W. C. Backer, J. Vac. Sci. Technol. **6**, 255 (1969).
[5] K. E. McCulloch, C. R. Tilford, C. D. Ehrlich, and F. G. Long, J. Vac. Sci. Technol. A **5**, 276 (1987).
[6] *JANAF Thermochemical Tubes*, 2nd ed. (NSRDS-National Bureau of Standards 37, Washington, DC, 1971).
[7] T. Cebeci and P. Bradshaw, *Physical and Computational Aspects of Convective Heat Transfer* (Springer, New York, 1984).